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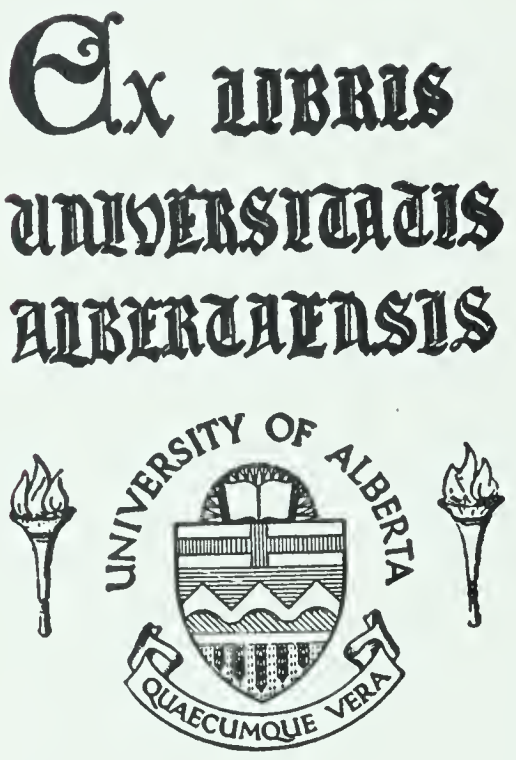

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THE UNIVERSITY OF ALBERTA

A HIGH TEMPERATURE X-RAY DIFFRACTION STUDY OF TITANIUM-ALUMINUM ALLOYS

A Thesis

Submitted to the Faculty of Graduate Studies

In Partial Fulfilment of the Requirements

for the Degree of Master of Science

DEPARTMENT OF MINING AND METALLURGY

by

ALBERT JOHN GOLDAK

EDMONTON, ALBERTA

SEPTEMBER 1960

UNIVERSITY OF ALBERTA

FACULTY OF GRADUATE STUDIES

The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for acceptance, a thesis entitled

A HIGH TEMPERATURE X-RAY DIFFRACTION STUDY

OF TITANIUM-ALUMINUM ALLOYS

submitted by ALBERT JOHN GOLDAK

in partial fulfilment of the requirements for the degree of Master of Science.

ABSTRACT

A high temperature specimen holder for a Norelco diffractometer, operable at 1000°C at 10^{-6} mm of Hg, is described.

Data are reported from X-ray diffraction patterns (taken at temperatures up to 900°C at 10^{-6} mm of Hg) of titanium alloys containing 15, 17, 20 and 25 atomic per cent aluminum. The results do not conform to the generally accepted constitutional diagrams nor can previously reported crystal structures be confirmed.

ACKNOWLEDGEMENTS

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INTRODUCTION

The results of several investigations of the titanium-aluminum system in the composition range 10 to 30 atomic per cent aluminum disagree on the identity of the phases present. This is principally due to the difference in purity of the alloys used by the investigators, for contamination at high temperatures (e.g. during melting and heat treatment) is difficult to prevent. The situation is made more vicious by the practice of long holding times at temperatures to attain equilibrium.

Earlier investigators[1] - [11] used miscroscopical metallography, X-ray diffraction, thermal analysis and electrical resistivity measurements to determine the phase boundaries in the titanium-aluminum system. With the exception of two high temperature X-ray diffraction studies of pure titanium, all investigators attempted to retain high temperature phases by rapid quenching. Thus, while phase boundaries have been determined by methods capable of yielding accurate results, the actual identification of the phases present is an open question. In view of the conflicting results and questionable identification of phases, this project undertook to clarify the situation by a high temperature X-ray diffraction method. Here the alloy sample is analysed at temperature, its crystal structure at temperature is determined, and the possibility of phase changes occurring during quenching is therefore avoided.

LITERATURE REVIEW

The first investigation of the entire titanium-aluminum system was carried out by Ogden et al.[1] in 1952. Alloys prepared from iodide titanium (99.95% Ti) and high purity aluminum (99.99% Al) were arc-melted in a water-cooled copper hearth.

"Alloy constitution was determined by metallography supplemented by X-ray diffraction. For a few alloys, transition temperatures were checked using the inverse rate method of thermal analysis. Melting temperatures of titanium-rich ingots were obtained by observing the melting of an ingot with a hole drilled in it."

Ogden's phase diagram, shown in figure 1, contains two intermetallic compounds. The crystal structure of $TiAl_3$ was determined by Brauer[2] in 1938: space group D_{4h}^{17}, I4/mmm with a body-centred tetragonal structure cell, a = 3.89kx and c = 8.579kx. Ogden, who indexed TiAl on a tetragonal structure cell with a = 4.015 Å and c = 4.0625 Å at 43.25 per cent[A] Al, suggested that the structure was of the ordered CuAu or $L1_0$ type.

Later in 1952 Bumps, Kessler and Hansen[3] published the phase diagram shown in figure 2, which is generally considered to be more reliable than Ogden's. Alloys were prepared from iodide titanium

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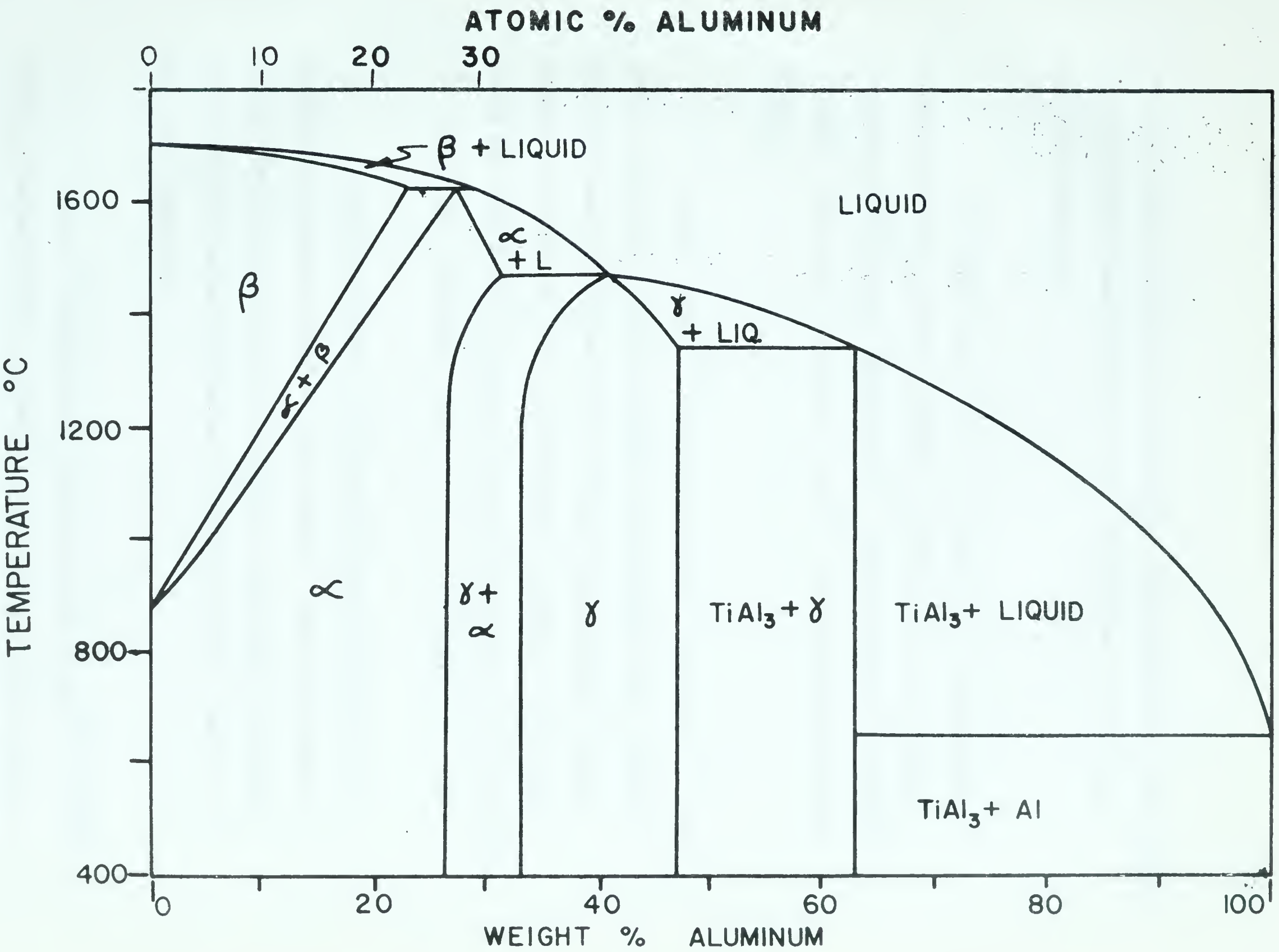
[A] Atomic per cent is used throughout this thesis.



Ti-Al Phase Diagram of Ogden et al

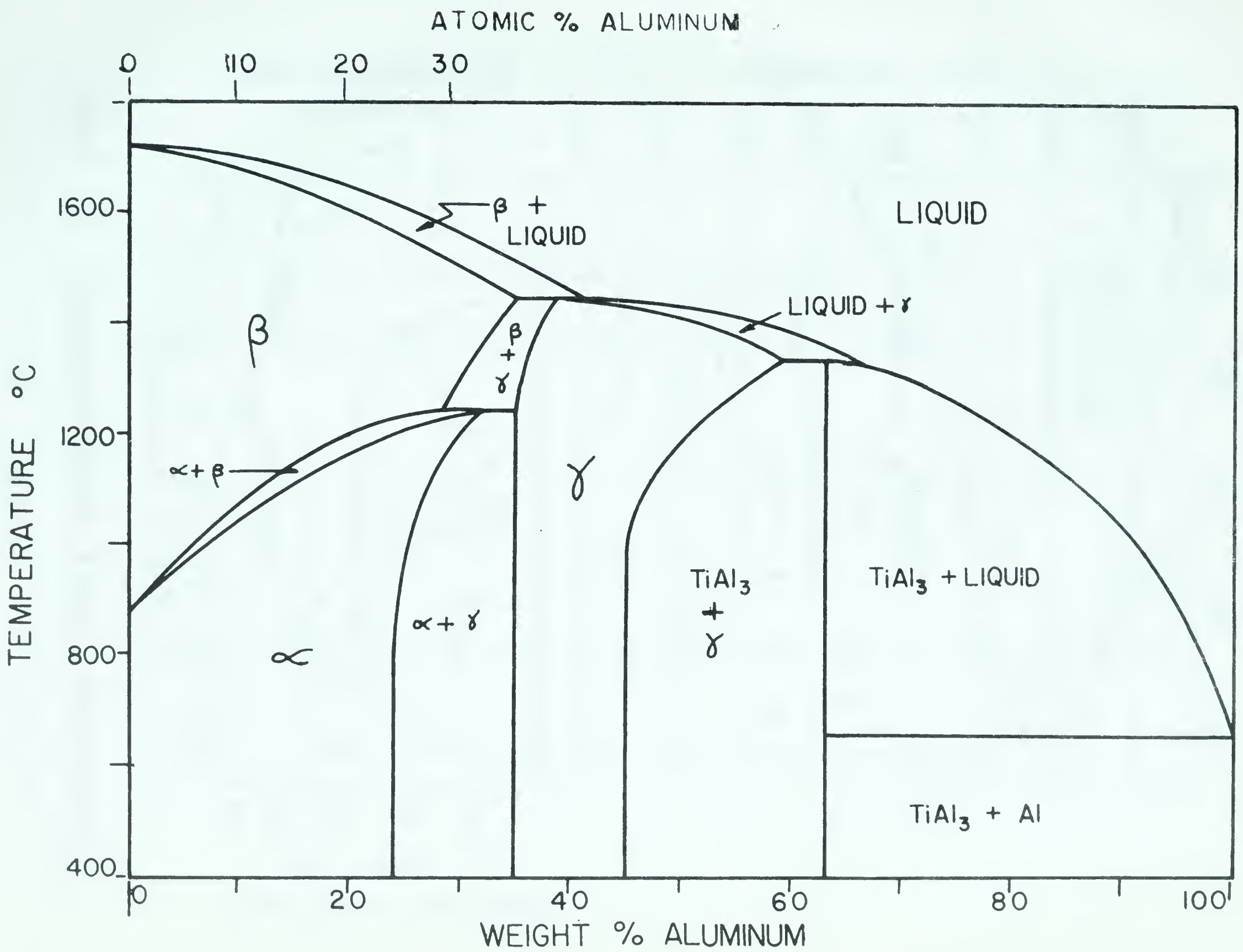
and high purity aluminum by arc-melting. Phase boundaries were located by microscopical metallography, X-ray diffraction, and incipient melting techniques.

The boundaries of the $\alpha + \beta$ field for titanium alloys containing 0 to 7 per cent aluminum, determined by the hydrogen pressure method by A.D. MacQuillan[4], confirm this part of Bump's diagram. The work of Kernilov et al.[5] also substantiates Bump's diagram.

In 1956, Sagel[6] reported electrical resistivity, magnetic, X-ray diffraction and microscopical metallography data (figure 3) for the range 0 to 45 per cent aluminum, that indicated two new phases in the region of 17 to 36 per cent aluminum. While Sagel could not detect the α_2 phase by X-ray diffraction, he did index the ε phase on a tetragonal cell with $a = 3.76$ Å and $c/a = 1.26$.

Clark and Terry[7] also reported X-ray evidence of an ordered Ti_3Al phase formed at 400 to 600°C: "X-ray photographs of alloys immediately preceding the ordered region show a characteristic and very marked broadening of the reflection". However, no data were reported.

Ence and Margolin have written several papers[8],[9],[10] on the titanium-aluminum system that are of questionable value. Their latest phase diagram, shown in figure 4, contains the phases



Ti-Al Phase Diagram of Bumps et al

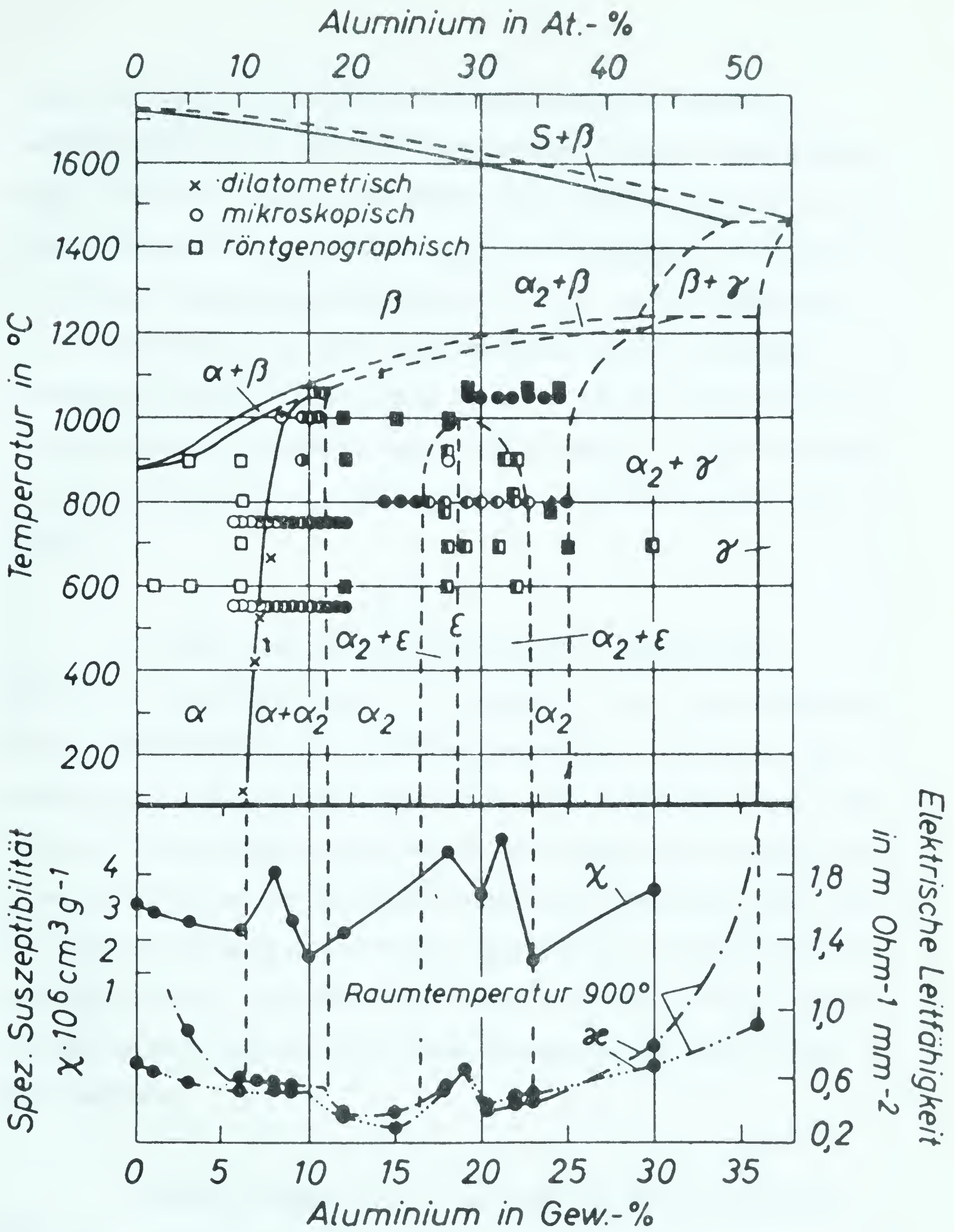


Bild 17. Zustandsbild Titan-Aluminium

FIGURE 3

Ti_2Al and Ti_3Al. It is difficult to understand why a phase is designated Ti_3Al when the composition of this phase is almost exactly Ti_5Al. Nor is it clear why an ordered A_2B structure (Ti_2Al) is said to be isomorphous with an ordered A_3B structure.(Mg_3Cd). While Ence and Margolin state that the structure of Ti_2Al has been established, neither they nor any of their four references (with the possible exception of Arderko et al., whose paper has not yet been made available) mention any attempt to analyse the structure. If the structure of Mg_3Cd and Ti_2Al are in fact isomorphous, this can and should be proved.

In 1959, Saulnier and Croutzelles[11] obtained more evidence of an ordered phase in the region of 17 per cent aluminum by electron micrography and microdiffraction methods. At 12 per cent aluminum an α solid solution was formed. The experimental data from alloys of 17 and 25 per cent aluminum were interpreted to suggest the presence of ordered and disordered domains with dimensions about 50 Å. An important characteristic of this investigation is that observations were obtained of a relatively small number of atoms - of the order of 10,000. A very large number of atoms are observed by the customary X-ray methods.

The work of Sagel et al., of Terry and Clark, of Ence and Margolin, of Saulnier and Croutzelles suggests that the α phase region of the diagram proposed by Bumps et al. is incorrect. However, there is insufficient agreement among the results of these

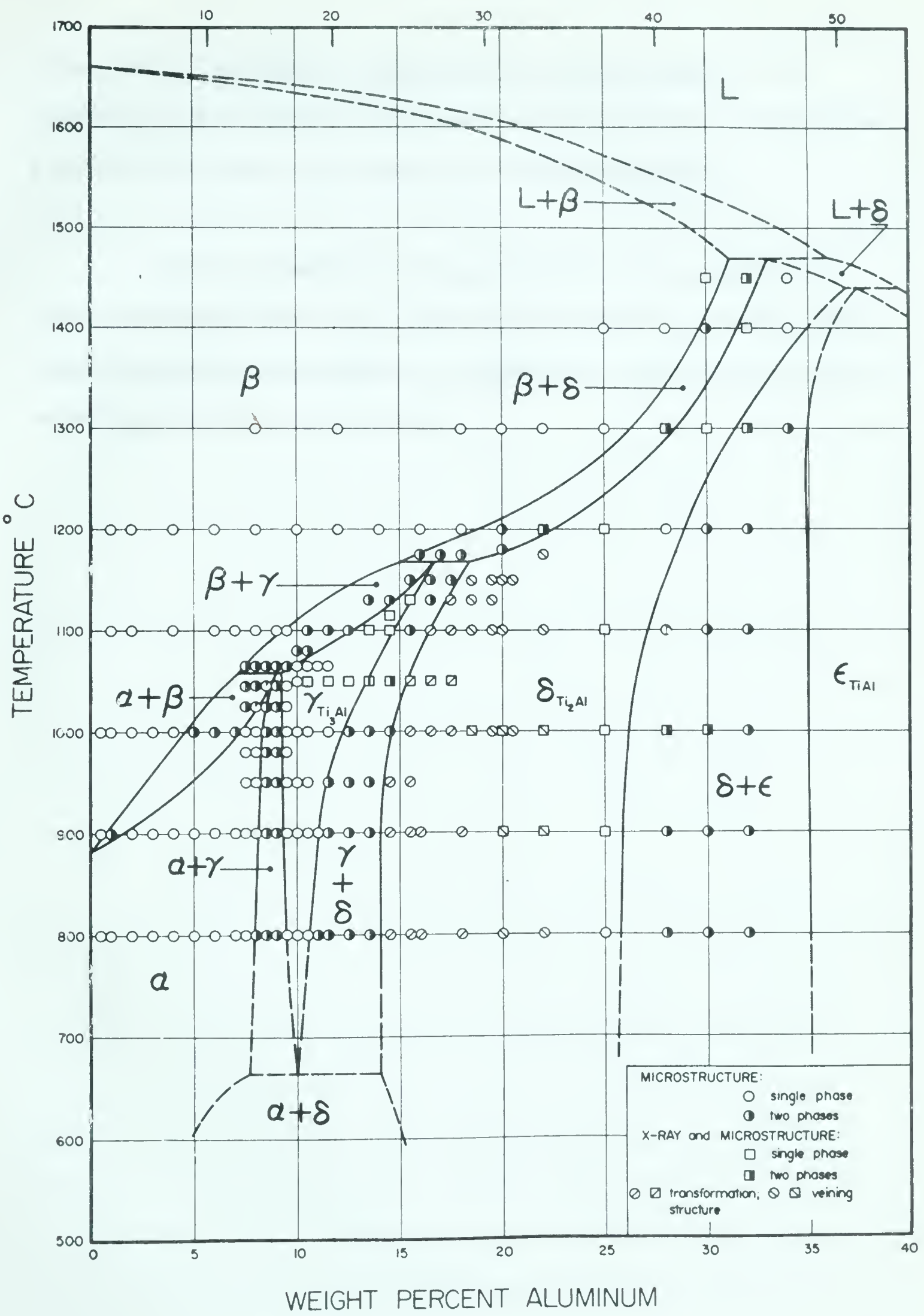


Figure 4 Partial Titanium-Aluminum Phase Diagram

investigators to permit a reconstruction of the diagram. The problems are the identification and structure analysis of the phases present and a study of the relations between the phases.

If the situation is to be clarified, it is necessary to devise techniques that incur less contamination of specimens, and to make experimental measurements at temperature, rather than to rely on retaining structures by quenching.

HIGH TEMPERATURE X-RAY DIFFRACTION APPARATUS

The first phase of this project was the design and construction of a high temperature X-ray diffraction apparatus. In order to obtain a meaningful X-ray diffraction pattern it is necessary (a) to measure and control the temperature of the specimen, (b) to know the composition of the specimen at all times, and (c) to satisfy the requirements of X-ray optics. A literature survey disclosed only one camera[12] and two specimen holders for a diffractometer[13],[14] that satisfied our basic requirements. The camera was rejected because of its complex design and the difficulty of making specimens. Further, a diffractometer is a more flexible instrument for reconnaissance studies. Consequently, it was decided to build a high temperature specimen holder based on a design by Chiotti[13], that could be attached to the department's North American Philips Diffractometer[A].

The plans of the specimen holder are shown in figures 7, 8 and 9. By comparison with Chiotti's design, shown in figures 5 and 6, it will be noted that, while the overall arrangements of components are much the same, almost all details have been changed. This is largely due to recent developments in vacuum equipment. The back plate and cover, which are machined from an 8" diameter round bar of

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[A] North American Philips designates the instrument a goniometer -- contrary to the convention of the International Union of Crystallography.

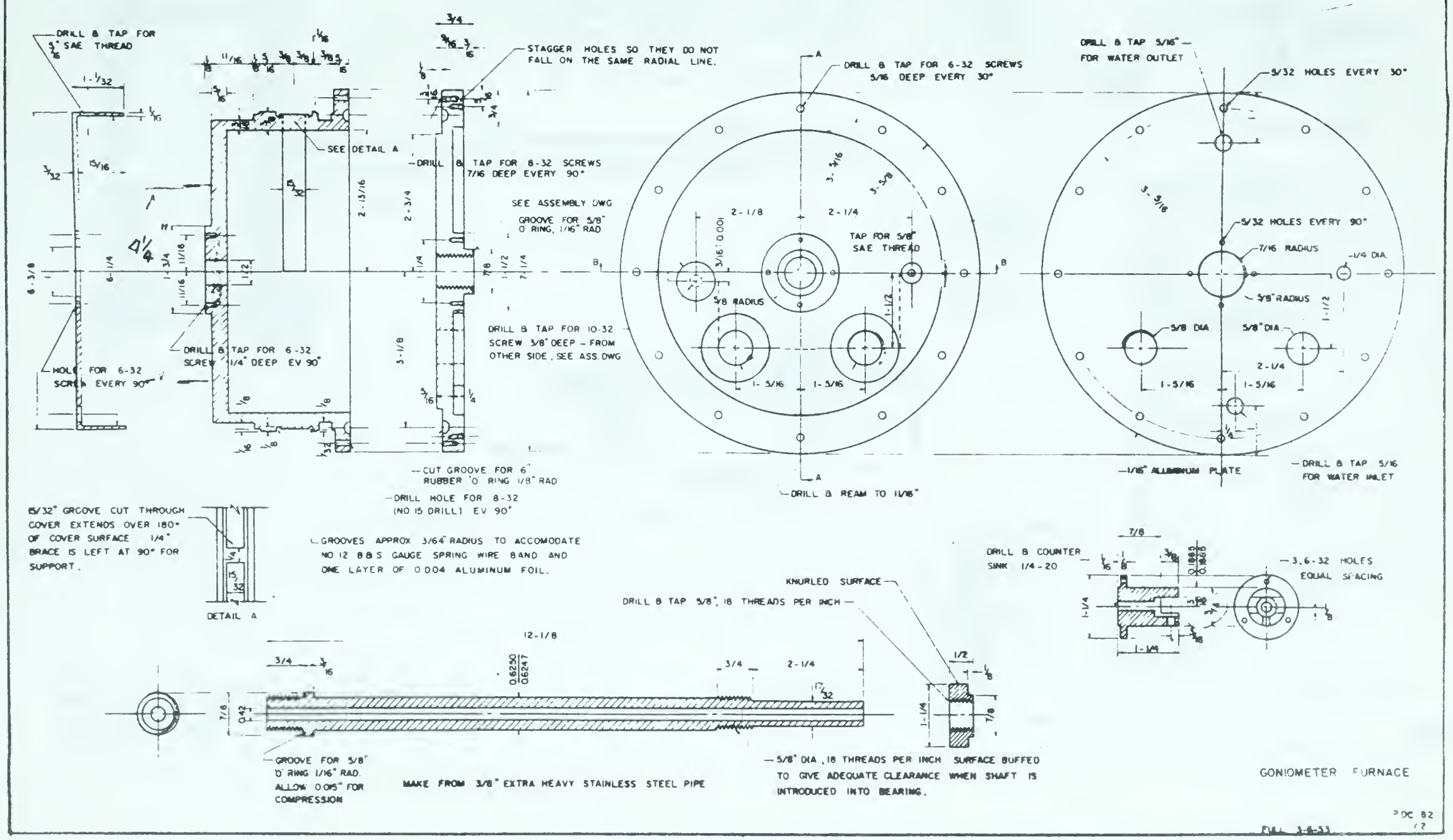

Figure 5 - Construction Details of Goniometer Furnace.

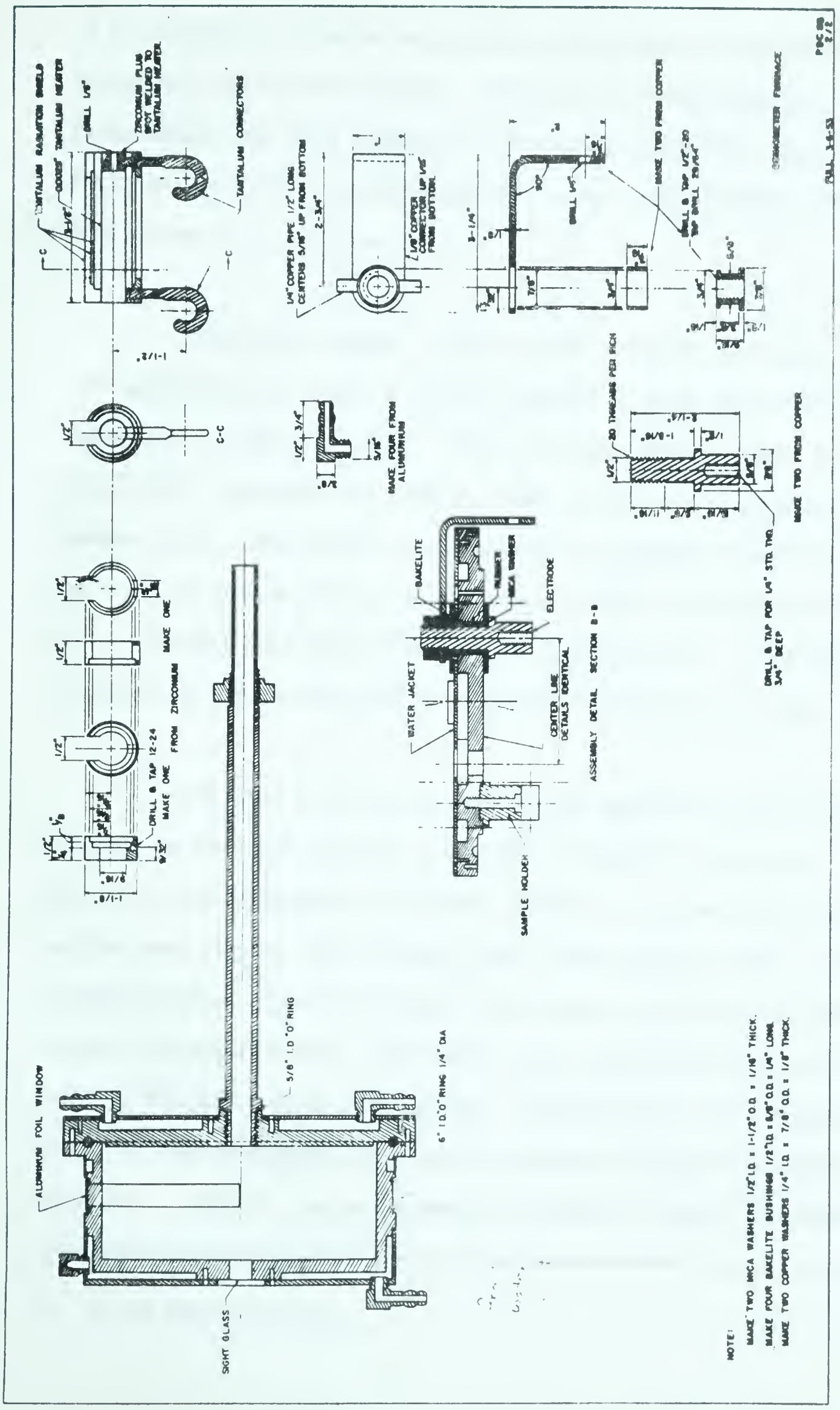

Figure 6 - Construction and Assembly Details for Goniometer Furnace.

17 ST aluminum, are water-cooled by jackets that are fastened with screws and sealed with Glyptal (although it is now apparent that "O" rings would have been superior). The steel shaft that replaces the shaft on the Norelco specimen holder screws into the back plate and is held firmly by a lock nut.

The X-ray window, a 12" x 0.75" x 0.020" beryllium sheet[A], is sealed against a 7¼" x 7½" "O" ring by a steel band, which resembles a large hose clamp. The beryllium sheet is more robust than the 0.0025" aluminum foil used by Chiotti and is not susceptible to vacuum leaks. The window can be heated to whatever temperature the "O" ring or vacuum grease can withstand (165°F for neopreme "O" rings) and is capable of transmitting 80% of incident Cu K radiation compared to 20% transmitted by a 0.025" aluminum foil window.

The most troublesome part of the apparatus was a suitable furnace to heat the specimen uniformly. Chiotti's design was rejected because large temperature gradients occurred. In our first design the alloys were cast as 3/8" diameter half round bars 3" long. A bar was clamped between copper electrodes and heated by passing an electric current through the bar. The X-rays were diffracted from the flat side of the bar. High temperatures, limited only by the softening point of the specimens, were easily obtained with good vacuum characteristics. However, large temperature gradients (200°C at 1000°C)

A Brush Beryllium Corp.

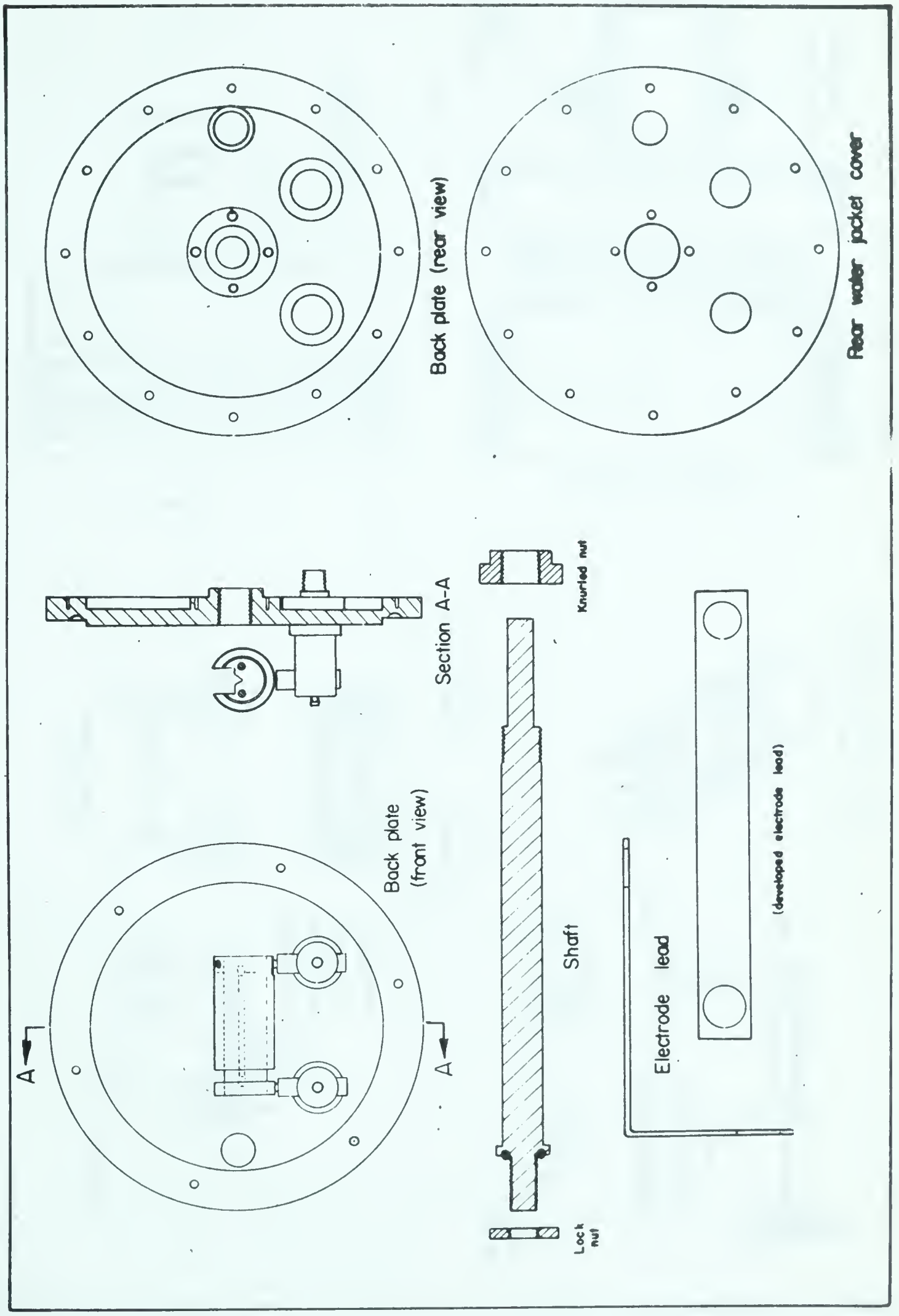
Back plate (rear view)
Rear water jacket cover
Section A-A
Knurled nut
Back plate (front view)
A
A
Shaft
Electrode lead
(developed electrode lead)
Lock nut

FIGURE 7

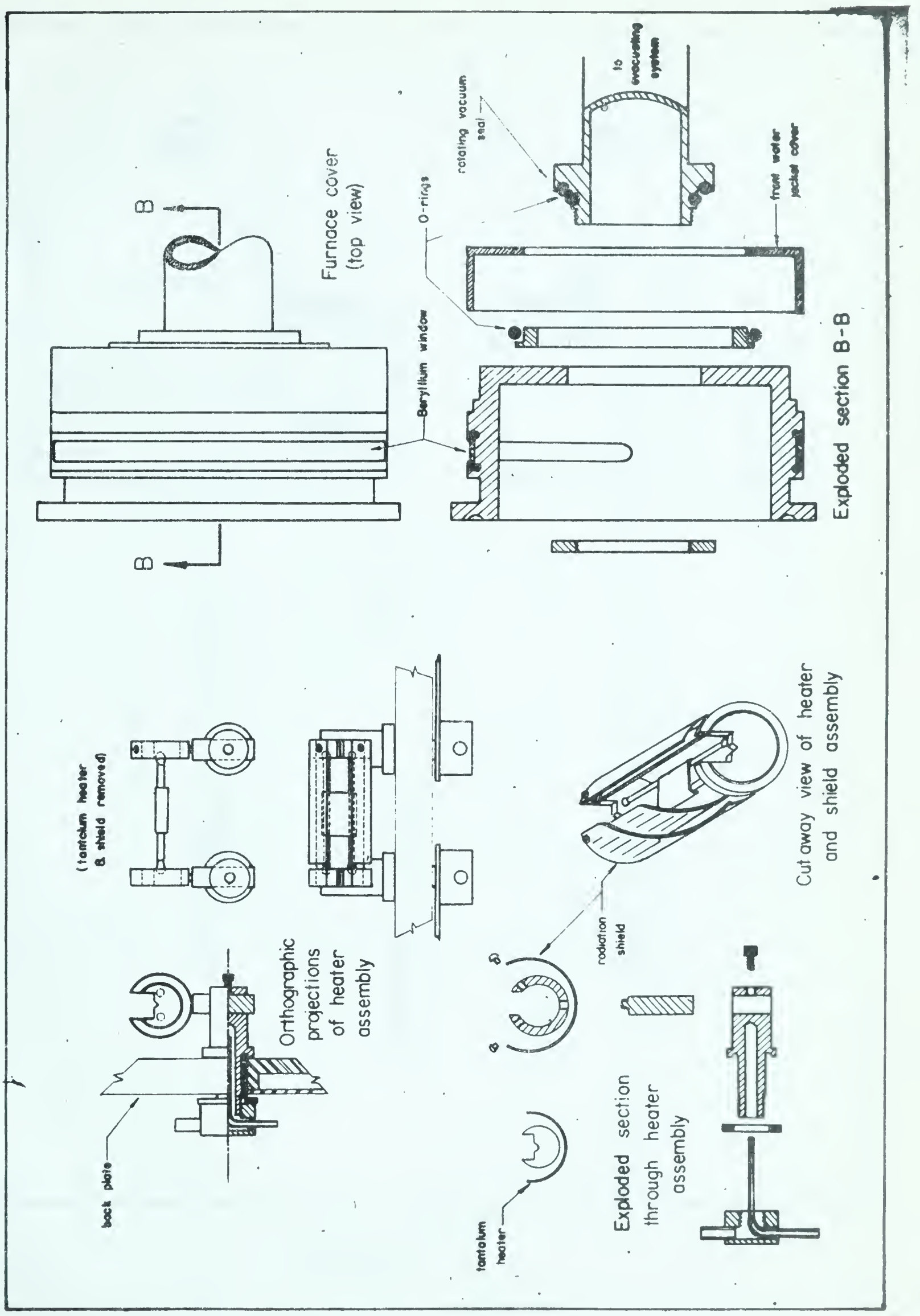
Furnace cover
(top view)
B
B
Beryllium window
O-rings
rotating vacuum seal
to evacuating system
front water jacket cover
Exploded section B-B
(tantalum heater & shield removed)
back plate
Orthographic projections of heater assembly
tantalum heater
Exploded section through heater assembly
radiation shield
Cut away view of heater and shield assembly

FIGURE 3

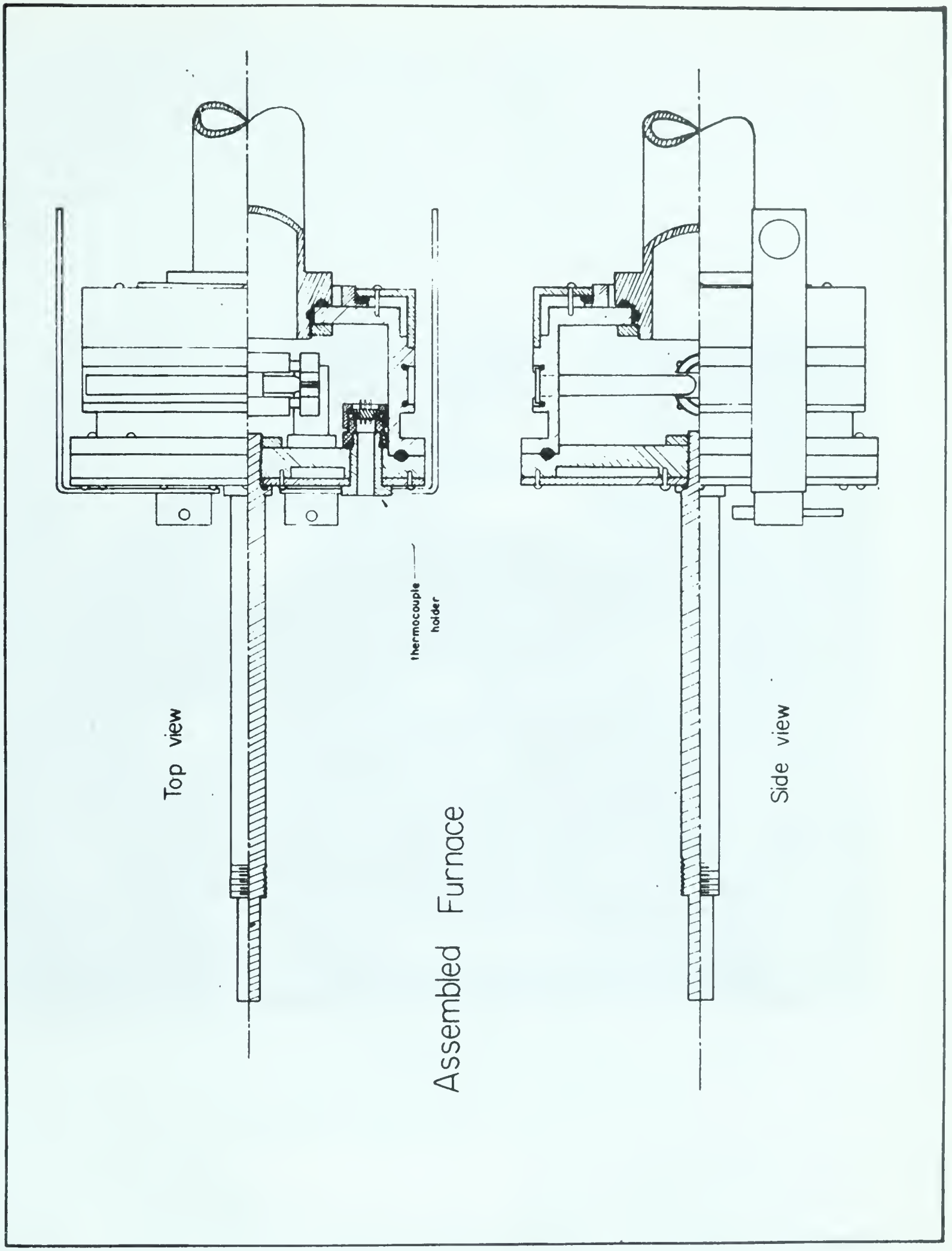

FIGURE 9

FIGURE 10

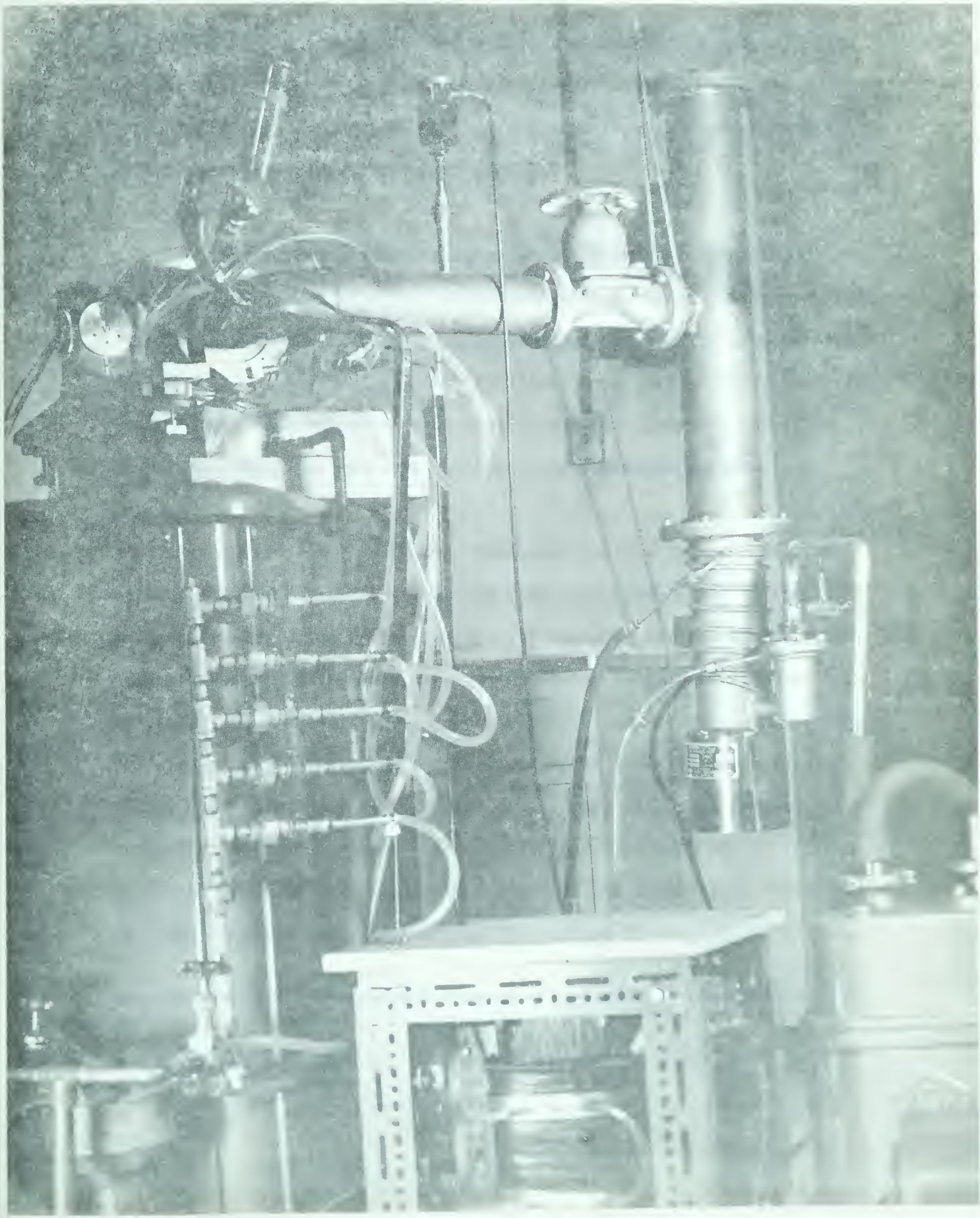

FIGURE 11

restricted the usefulness of this method. An attempt to reduce the temperature gradients by varying the cross-sectional area of the specimen failed. It is felt that this design might be useful at very high temperatures, possibly above 1500°C.

The next design is illustrated in figures 7, 8 and 10. A tantalum sheet[A], 0.002" thick, is placed between copper bushings, which are attached to the electrodes by round copper bars. The specimen is placed on a tantalum sheet, which is stretched across two tungsten rods. The tungsten rods fit into small quartz tubes in the inner copper bushings. Thus, the specimen and stage are thermally isolated.

Power from a 220 volt "Stabline" voltage regulator is fed through a 2.5 KVA "VARIAC" to a 1:30 step-down transformer to the furnace element. The voltage across the element, and consequently the temperature of the specimen, are controlled by varying the primary voltage by the variac. The step-down transformer was made by replacing the high voltage winding of a discarded 10 KVA 2200/220 volt transformer with four turns of 1" x 1/8" cooper bar. All high current conductors are water-cooled. At a specific variac setting the voltage regulator provides ± ½% regulation, which causes temperature variations of ± ½%, assuming the resistance of the furnace is constant.

A Murex Ltd., England.

Temperature was measured by two thermocouples placed approximately 1 cm apart on the specimen. At a specific variac setting the temperatures measured did not differ by more than 30°C nor did the temperature measured fluctuate more than $\pm$ 1°C.

At high temperatures the specimen can be kept in an inert gas atmosphere, or a vacuum, to avoid contamination. The most suitable inert gas is helium, since it permits a maximum intensity of transmitted X-rays. Because one atmosphere of inert gas, 99.999% pure, contains foreign molecules equivalent to a vacuum of 10^{-4} mm of Hg, it was felt that less contamination would be incurred in a vacuum of 10^{-6} mm of Hg than in an inert atmosphere. A study of the kinetics, which would be very difficult, might lead to a different conclusion. While such a vacuum is easily obtained in a metal system with "O" ring seals, it is necessary to be able to bake the entire system at a temperature about 400°C to achieve better vacua. This is not practical in a complex system.

The high vacuum system - a 300 litre/sec oil diffusion pump, cold finger, 2" valve and vacuum lines - is suspended from a roller on a 4" x 4" I beam placed on the ceiling joists.(figure 11). Thus the high vacuum system moves easily in all directions so that very little stress is placed on the diffractometer. The high vacuum system is attached to the cover of the specimen holder by a rotating "O" ring seal. It was feared that the gears in the diffractometer would not be strong anough to rotate the specimen holder under vacuum because

of pressure on the seal. However, the seal can be rotated one degree per minute by an extremely small torque.

In order to achieve high pumping rates it is necessary to use large diameter vacuum lines. The smallest cross-sections in this system are a two inch vacuum valve and two sections of 2" diameter pipe 2" long. In contrast, Chiotti's system was evacuated through a hollow steel shaft $^{3}/_{8}$" i.d. x 12" long that replaced the Norelco specimen holder shaft. Vapour in the system condenses on the cold finger, which is filled with dry ice and alcohol, or with liquid air. The vacuum pressure, which is measured by a Balzers ionization gauge, is easily maintained at 2×10^{-7} mm of Hg ($20^{o}C$) and 7×10^{-7} mm of Hg ($1000^{o}C$) when Apiezon "B" diffusion pump oil is used. Presumably lower pressures could be obtained with Apiezon "C" diffusion pump oil. It was not possible to detect any leak in the system with a VEECO helium leak detector.

Armco iron was chosen to test the apparatus. The transformation temperature was measured at 870 to $890^{o}C$ compared to the value of 900 to $910^{o}C$ usually given. The parameters of α at $20^{o}C$ and γ at $950^{o}C$ were 2.865 Å and 3.647 Å respectively compared to values of 2.866 Å and 3.656 Å given by Barrett[15] or 2.860 Å. and 3.6394 Å given by Pearson[16]. The γ parameter of 3.647 Å is probably not accurate because the highest angle line observed was 44^{o} θ.

EXPERIMENTAL METHOD

Alloys were prepared from iodide titanium and spectrographic aluminum by levitation melting in an argon atmosphere[17]. The alloys were quickly heated above the melting point, where high frequency eddy currents insured a homogeneous alloy, and chill cast in split copper moulds. The alloy was molten for only a few seconds compared to the several minutes required in the arc-melting technique. The nominal compositions were 15, 17, 20 and 25 per cent aluminum. The weight lost during melting was not more than 0.1 per cent.

It is difficult to reduce a titanium-aluminum alloy containing less than 30 per cent aluminum to a powder for diffraction studies. If the alloy is filed with a steel file the resulting powder might contain as much as 1% iron. An alternative method is to heat the alloy in a hydrogen environment, crush the resulting alloy, and then remove the hydrogen from the powder by heating in a vacuum. This method requires rather elaborate apparatus and probably permits some contamination. As a last resort solid specimens were used, even though preferred orientation reduces the value of relative intensity data. These specimens, approximately 12 mm in diameter and 2 mm thick, were cut from the centre of the ingot with a diamond saw.

An X-ray diffraction pattern was run on the as-cast specimen at room temperature while the specimen holder was being evacuated. When the pressure reached 7×10^{-7} mm of Hg, the temperature was slowly

raised to 600°C and a second diffraction pattern taken. Then two to four patterns were taken at 900°C over a period of about 12 hours. After cooling to room temperature, the last diffraction pattern was taken. Examples of the data from these patterns are given in tables I, II and III. Copper K radiation and a nickel filter were used throughout this investigation.

The structure cells were determined by trial and error. As all patterns were indexed on hexagonal cells, the appropriate formula is

$$\frac{\lambda^2}{4 \sin^2\theta} = \frac{4}{3} \cdot \frac{h^2 + hk + k^2}{a^2} + \frac{\ell^2}{c^2}$$

It is seldom appreciated that indexing a pattern on a specific cell does not necessarily establish the crystal lattice. In this case the crystal lattice could be rhombohedral.

The amount of contamination incurred was checked by measuring the hardness of an iodide titanium specimen before and after a run. After 12 hours at 600°C at 10^{-6} mm of Hg the surface hardness measured with a Tukon tester (standard diamond and 1 Kg load) showed a decrease from 87 to 82. After 12 hours at 600°C followed by 3 hours at 900°C the hardness decreased from 100 to 76. Presumably the decrease is associated with the effect of annealing. It appears that the contamination of the surface from which X-ray data were obtained was negligible.

There is further uncertainty in the composition of the

specimen because the rate of sublimation of aluminum is probably faster than the rate of sublimation of titanium, and this would decrease the aluminum content at the surface of the specimen.

It is difficult to estimate how much the rates of sublimation would be reduced in an inert gas atmosphere, where the possibility of a metal atom in the gaseous state returning to the specimen is increased. In both inert atmosphere and in vacuum the metal atom will be deposited on the walls of the container. Further, convection currents (due to the temperature gradients in the specimen holder) will prevent the establishment of a static blanket of helium immediately above the specimen.

It would be difficult to measure this effect quantitatively. The error is expected to be systematic and therefore not of major importance in this investigation.

TABLE I

Specimen - 15 per cent aluminum

Temperature - $600^{o}C$

Pressure - 10^{-6} mm of Hg

Copper K radiation

Results taken from one pattern

Structure cell - hexagonal - $a = 2.94$ Å $c = 4.70$ Å

hkl	observed $\sin^2\theta$	calculated $\sin^2\theta$	Relative Intensities
100	0.0918	0.0918	7
002	0.1077	0.1076	19
101	0.1180	0.1187	33
102	0.1983	0.1994	.6
110	0.2720	0.2757	3
103	0.3331	0.3339	100
200	0.3657	0.3676	4
112	0.3814	0.3833	9
203_1	0.6057	0.6087	54
203_2	0.6088	0.6117	
210_1	0.6321	0.6423	.2
210_2	0.6395	0.6454	
105_1	0.7599	0.7631	.2
105_2	0.7626	0.7668	
300_1	0.8241	0.8258	1.3
300_2	0.8285	0.8298	

TABLE II

Specimen - 17 per cent aluminum

Temperature - 900°C

Pressure - 10^{-6} mm of Hg

Copper K radiation

θ values are averaged from four patterns

Structure cell - hexagonal - a = 2.96 Å c = 4.69 Å

hkl	observed $\sin^2\theta$	calculated $\sin^2\theta$	Relative Itensity
100	0.0898	0.0904	2
101	0.1168	0.1174	10
102	0.1956	0.1984	1
110	0.2692	0.2712	4
111	0.3046	0.2982	4
103	0.3298	0.3334	100
113	0.5209	0.5142	1
210	0.6331	0.6328	18
212	0.7429	0.7411	7

TABLE III

Specimen - 25 per cent aluminum

Temperature - 20°C

Pressure - 10^{-7} mm of Hg

Copper K radiation

Previously the specimen had been held at 450°C for 24 hours.

Results are taken from one pattern.

Structure cell - hexagonal - $a = 5.77$ Å $c = 4.69$ Å

hkl	observed $\sin^2\theta$	calculated $\sin^2\theta$	Relative Intensity
100	0.0189	0.0239	1
101	0.0514	0.0508	3
200	0.0950	0.0952	27
002	0.1107	0.1080	3
201	0.1227	0.1222	100
112	0.1810	0.1784	7
202	0.2048	0.2032	3
300	0.2132	0.2142	1
220	0.2834	0.2856	3
302_1	0.3199	0.3217	3
302_2	0.3217	0.3233	
203	0.3420	0.3382	2
400_1	0.3773	0.3802	23
400_2	0.3790	0.3820	
402	0.4871	0.4888	24
421	0.6873	0.6934	21
224_1	0.7216	0.7164	40
224_2	0.7252	0.7200	

DISCUSSION

The diffraction lines in all patterns obtained in this investigation were as wide as 30'θ at half peak height compared to 5'θ from a silicon standard. The variation in width of diffraction lines in a specific instruments, such as the department's diffractometer, is due to the variation in the number of planes of atoms that act together to diffract the X-ray beam. Only those atomic planes contained in a volume of the crystal that is relatively perfect, in the sense that the periodicity is perfect, act together to diffract X-rays.

The accuracy of the cell parameters is limited by the accuracy with which the angle θ is measured. If the lines are as broad as they were in this investigation it is generally considered that the accuracy of the cell parameters will be of the order of one per cent, compared to the 0.003% possible if the lines are narrow. As a result it is not possible to use the variation in lattice parameters with composition to detect phase boundaries.

Many types of imperfections, such as dislocations, stacking faults, domains of order and disorder, precipitates, contamination, and lattice distortion, might be present. The customary X-ray methods only give an approximate notion of the concentration of dislocations present. Stacking faults cause reflections from the plane of faulting to be very asymmetric - a condition not observed in this work. The effects of lattice distortion or contamination are difficult to assess in this case.

Ordered and disordered domains possess some similar features to precipitates. Ordering, which refers to a specific type of crystal structure, causes superlattice reflections. A precipitate, which refers to a mode of occurrence of a crystal structure, need not be ordered. If the ordered domains are small, (Saulnier's[11] paper indicates a domain dimension of 50 Å), then the superlattice reflections that are inherently weak would be broad and very difficult to detect. If the lattice parameters of the ordered and disordered phases were sufficiently different, the fundamental lines would be resolved, as was observed in the specimen containing 20 per cent aluminum. This is an interesting possibility because the structure of the 25 per cent aluminum specimen appears to be an ordered modification of the close-packed hexagonal structure. This agrees with Saulnier's interpretation.

If the effects of contamination in this investigation are not significant, then the imperfections in crystal structure are a characteristic feature of the titanium-aluminum system. A study of the crystal texture or substructure by methods such as electron microscope metallography or divergent beam X-ray photography appears to be the best way to further our knowledge of the titanium-aluminum system.

SUMMARY

1. Specimens containing 15 and 17 per cent aluminum were indexed on hexagonal cells with $a = 2.94$ Å, $c = 4.70$ Å at 600°C and $a = 2.96$ Å, $c = 4.69$ Å at 900°C respectively. The crystal structures, which were probably close-packed hexagonal, were stable at 900°C for 12 hours.

2. In the 20 per cent aluminum specimen two phases existed that were indexed with $a = 2.96$ Å, $c = 4.69$ Å and $a = 2.95$ Å $c = 4.69$ Å. The two phases present are probably the hexagonal close-packed alpha phase and the hexagonal phase that is observed in the 25 per cent aluminum specimen - in which case one of the above "a" parameters should be doubled. In this alloy which had previously been held at 950°C for 42 hours, the crystal structure was stable at 900°C for 12 hours.

3. The specimens containing 25 per cent aluminum was indexed on a hexagonal cell with $a = 5.77$ Å, $c = 4.69$ Å. This structure was stable at 900°C for 12 hours.

4. The crystal structure of all specimens studied was very imperfect. While some stacking faults might be present, the line broadening is probably due to the small volume of perfect crystal.

5. While these results do not permit the construction of a constitutional diagram, it is clear that they do not conform to the data presented in the existing constitutional diagram.

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